



NASA Science Symposium for Increments 53 & 54

Advanced Colloids Experiment (ACE-M2R: Weitz/Lu – Harvard) Science Background



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ISS Increments 53 and 54 Science Symposium

Advanced Colloids Experiment

Microscopy Reflight (now with confocal) – ACE-M2R (Weitz / Lu – Harvard)

- Science Background and Hypothesis
- Investigation goals and objectives
- Measurement approach
- Importance and reason for ISS
- Expected results and how they will advance the field
- Earth benefits/spin-off applications



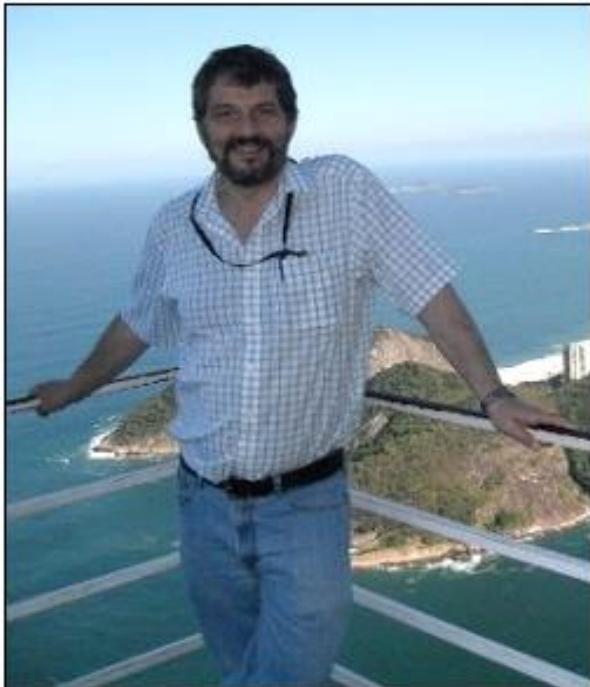
Advanced Colloids Experiment - Microscopy 2 Reflight (ACE-M2R)

Science Overview





Harvard ACE-M2 science team



Professor David Weitz



Dr. Peter Lu

Microgravity Colloids Program

Two Main Themes:

1. Answer fundamental science questions - understanding
2. Help lay the foundations for colloidal engineering – applications

The Advanced Colloids Experiment (ACE)

Science Objective (the big picture)

(Decadal Survey Area Complex Fluids, FP1 and AP 5):

ACE will remove gravitational jamming and sedimentation and then use a microscope to observe and help understand what happens to colloids at the particle level.

BCAT experiment on the ISS and NASA astronaut Suni Williams



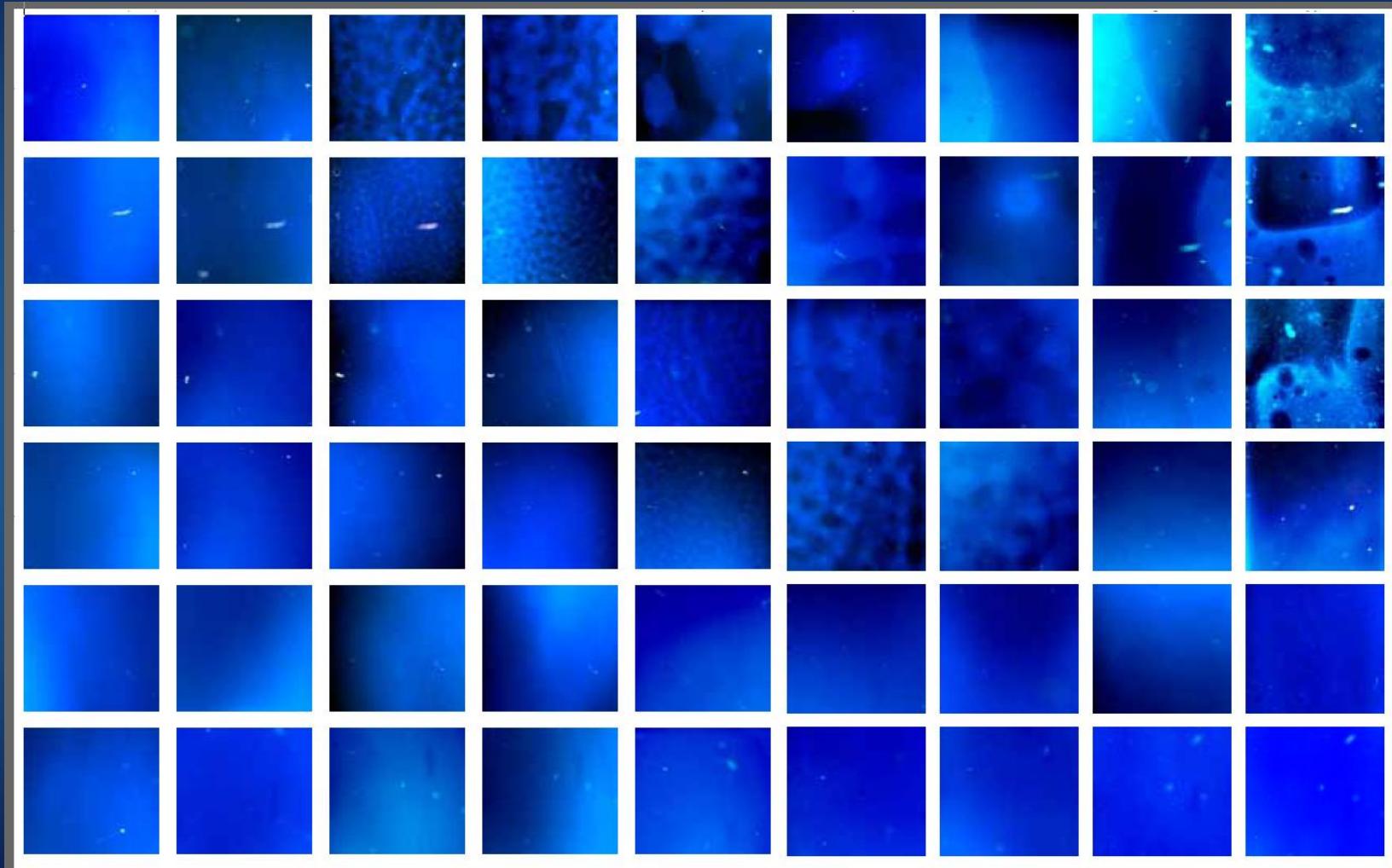
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Time evolution of model critical fluid samples in the BCAT-3 experiment.



Glenn Research Center

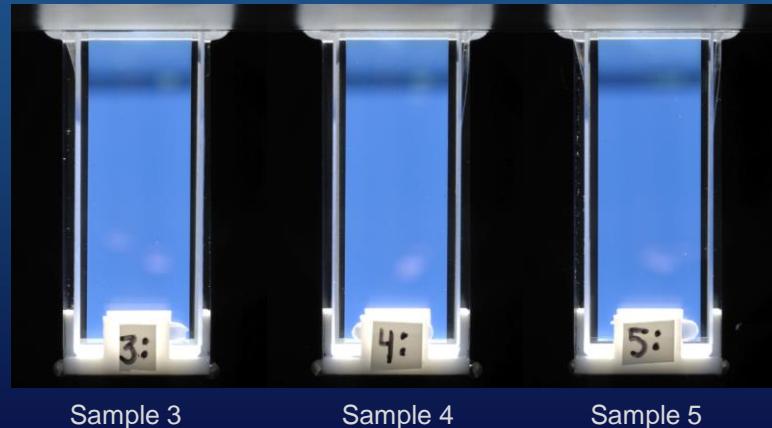
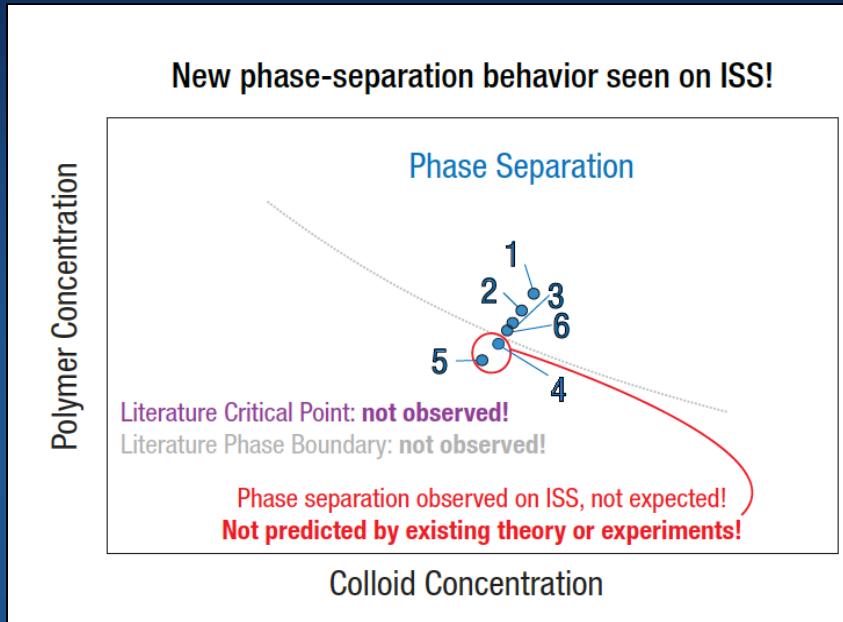
Samples



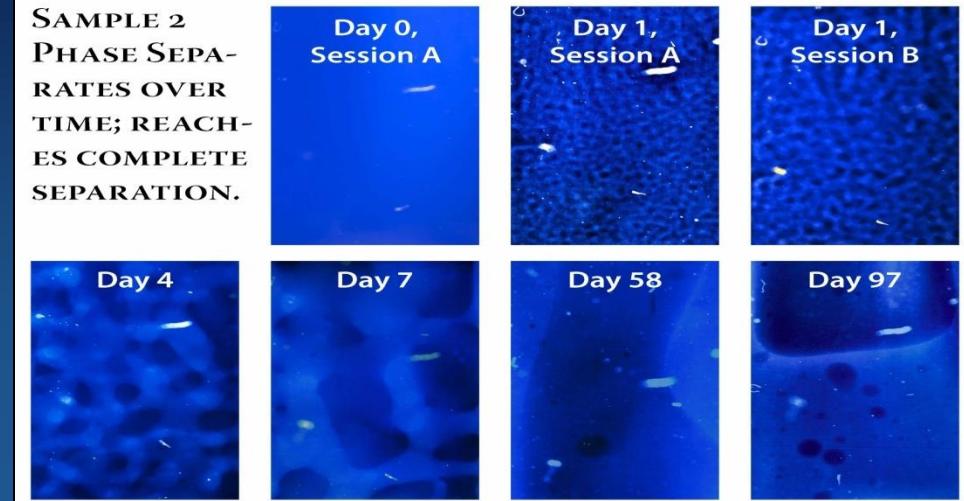
BCAT-6: Phase separation samples

David Weitz and Peter Lu - Harvard

Pinning down the critical point.



SAMPLE 2
PHASE SEPARATES OVER TIME; REACHES COMPLETE SEPARATION.



Colloid rich phase



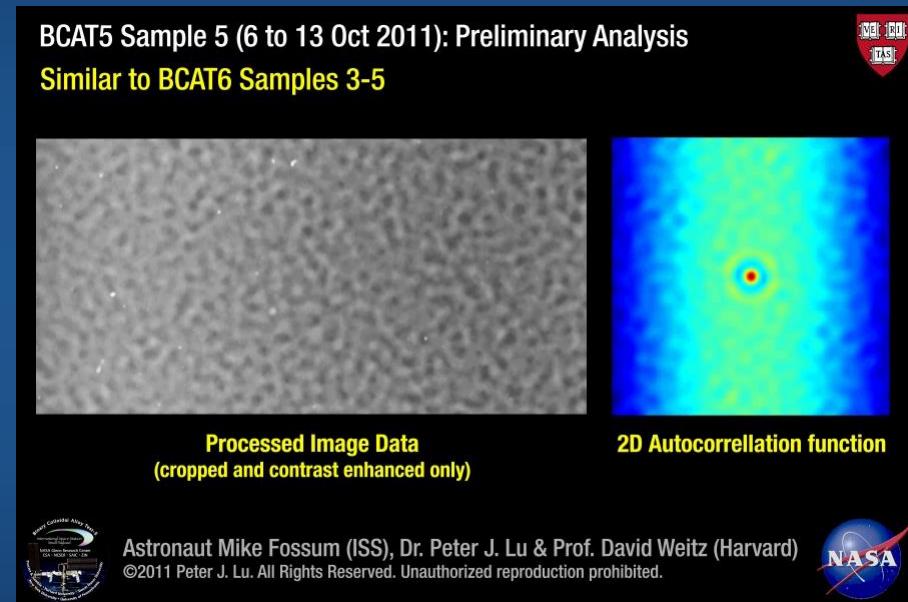
Colloid poor phase

The literature on these systems is wrong. This study provides the understanding for phase separation of *model* systems – also relevant for developing a basic model for product shelf-life.

New compositions had to be made after BCAT-3 and BCAT-4 results showed that others had been looking in the wrong place in the phase diagram for the critical point. These BCAT-6 samples are now hunting the critical point (with gravitational masking removed).

Movie of Phase Separation in μ -g

(BCAT-5, sample 5, October 6 - 13, 2011)



Phase separation no longer has a top or bottom in microgravity.
Coarsening can be observed over an extended period of days to months,
as seen in the above movies. Please click on images to play movies.

ACE-M2 flight data

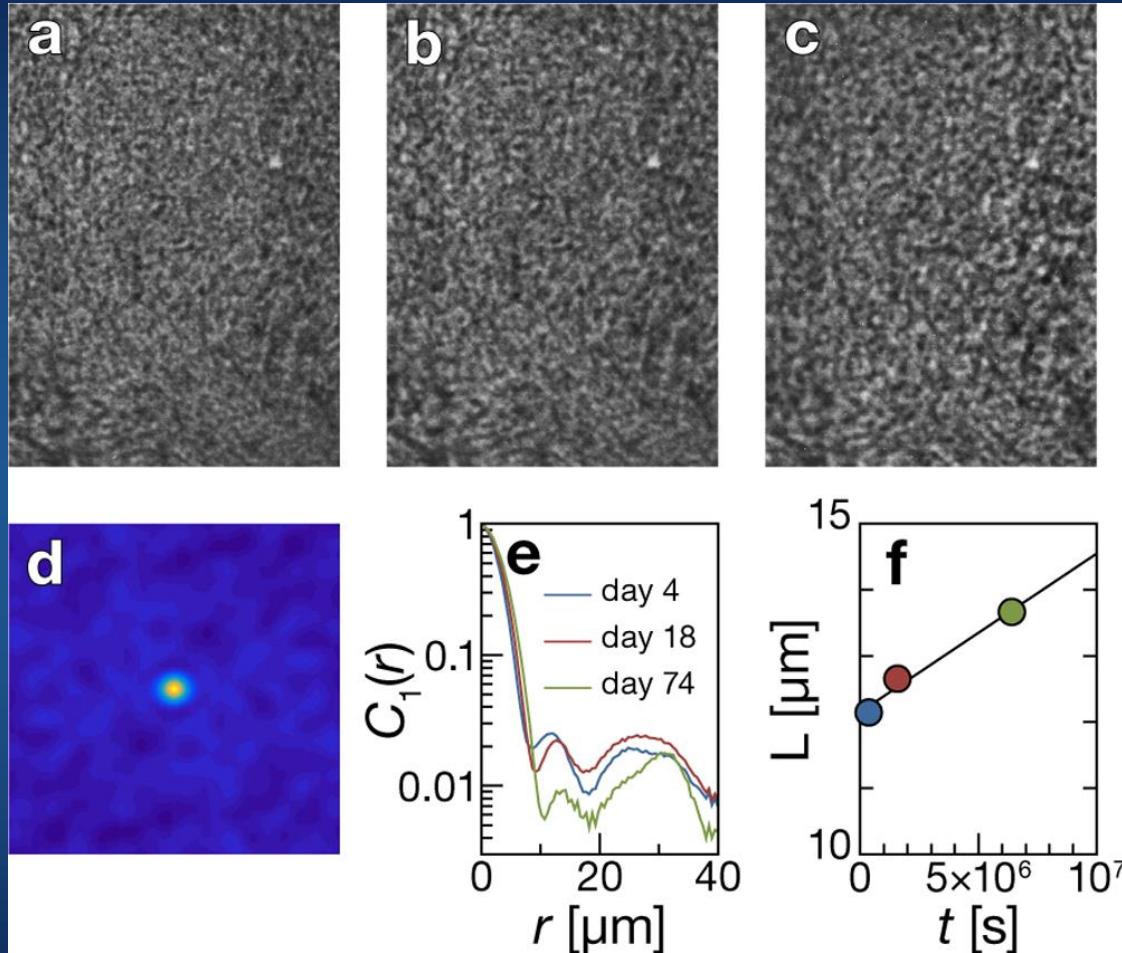


Figure 8. Phase-separating colloid-polymer sample in microgravity imaged using wide-field fluorescence microscopy with a $10\times$ objective, (a) 4, (b) 18, and (c) 74 days after initial mixing. The sample coarsens without fading or loss of contrast due to dye degradation or leakage.

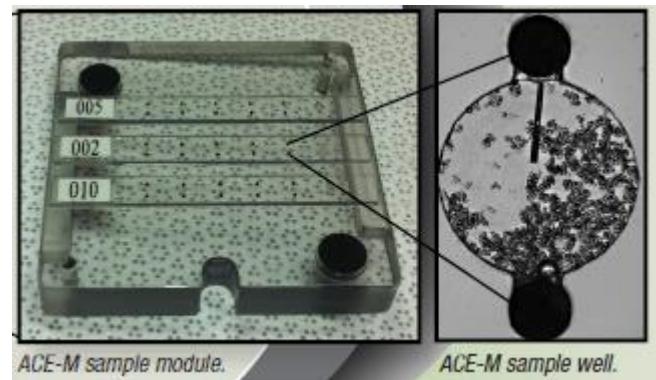
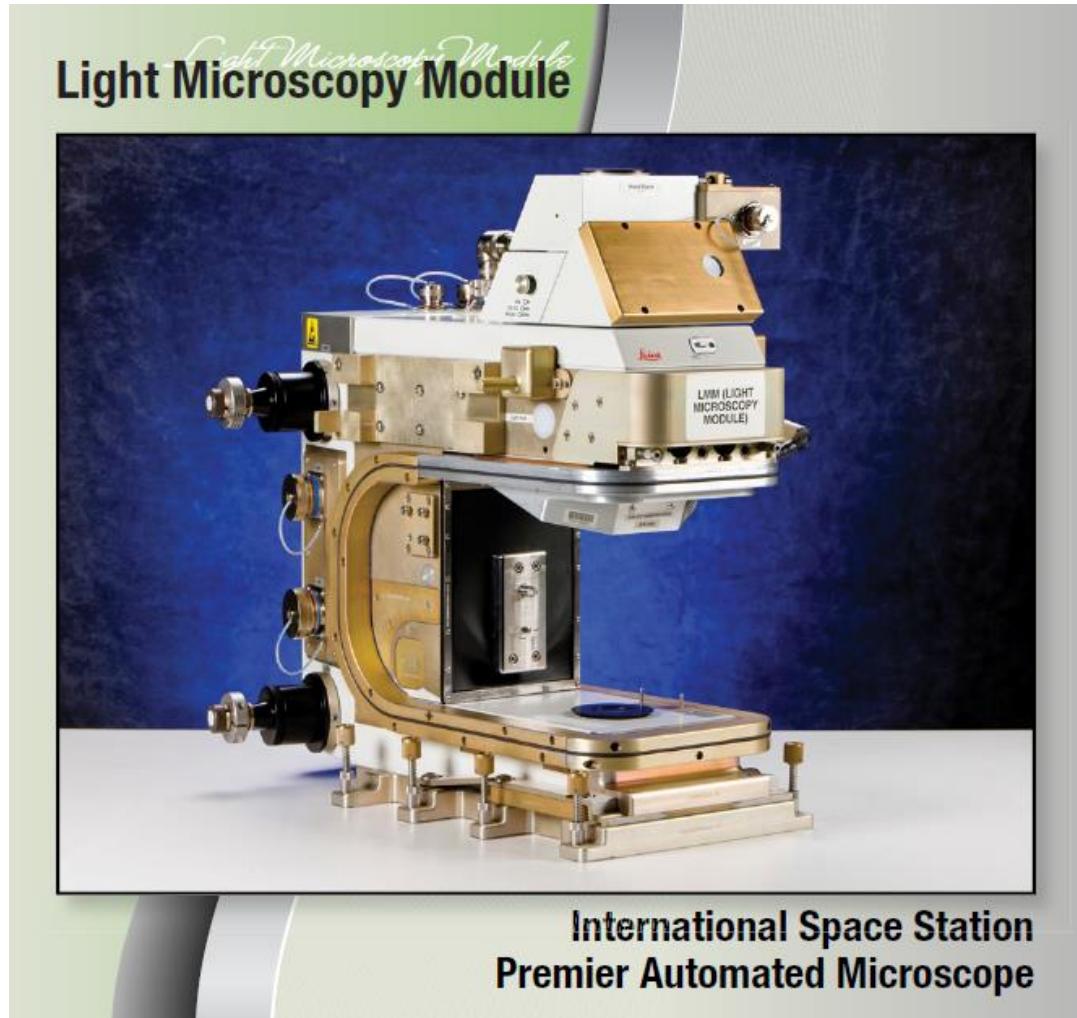
(d) 2D intensity autocorrelation extending ± 50 microns in \hat{x} and \hat{y} real-space dimensions. (e) 1D azimuthal averages at the times shown in (a–c). (f) Position of the first peak, corresponding to a characteristic length scale L , grows at 0.24 pm/s .

The epub citation for the above, which is ahead of print, is:
[Langmuir](#). 2017 Jun 15. doi: 10.1021/acs.langmuir.7b00852.



Advanced Colloids Experiment (ACE)-M2

And then the research tools got even better. And confocal awaits.





With LMM we can answer new science questions.

- The small ACE-M2R sample volumes allow the use of a 3-component solvent that both index matches and almost buoyancy matches the colloids (1000 times better than BCAT).
- This allows us to better locate the critical point.
- This also allows the rate of phase separation to be slowed down so that the early stages of spinodal decomposition can be observed – even 2 hours after the sample has been homogenized (mixed) and LMM is powered-up.



ACE-M2R: Investigation Goals and Objectives 2/2



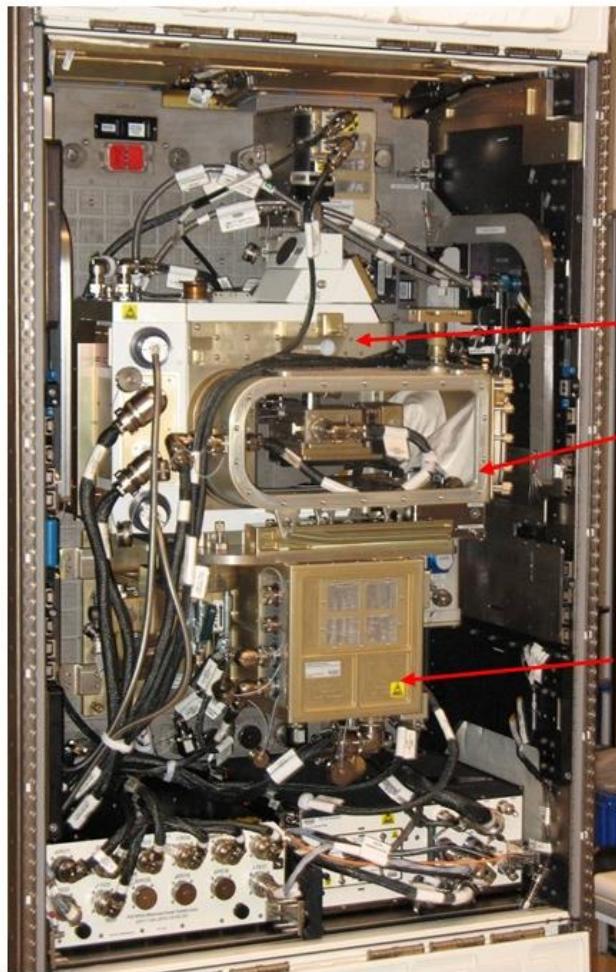
- Theory says that the rate of phase separation changes over time. How much and when? Growth of the domain size should exhibit a crossover from diffusion-limited dynamics to interfacial-tension-driven dynamics [A.E. Bailey, PRL **99**, 205701 (2007); E. D. Siggia, Phys. Rev A **20**, 595 (1979)].
- The Advanced Colloids Experiment (ACE) should be able to observe the very EARLY stages of phase separation (when the growing blobs were too small for the BCAT experiment to photograph them).
- Experiment guides us. “In theory, theory and practice are the same. In practice, they are not.” - Albert Einstein

Measurement approach – 1/16

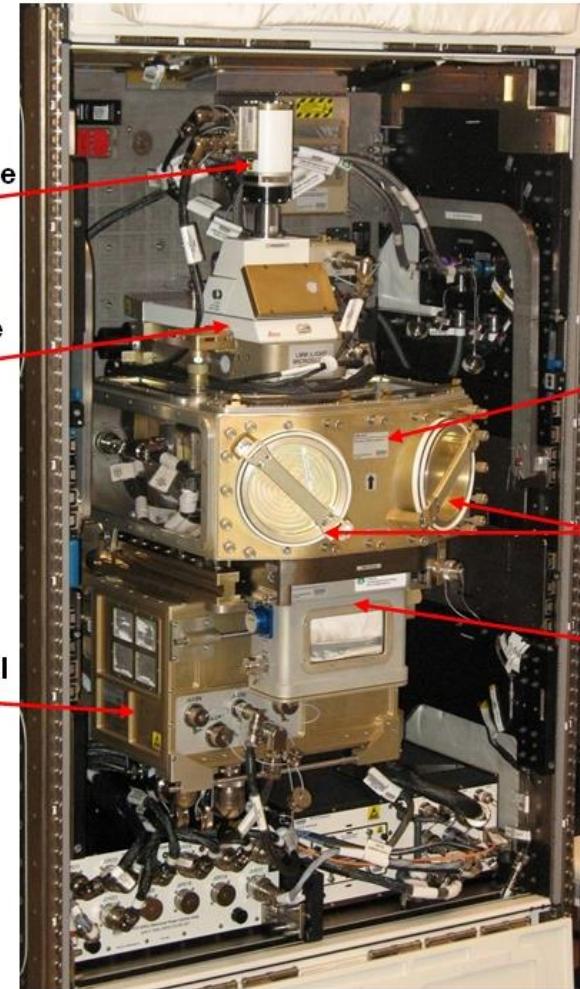
We will be using a flight-hardened Commercial-Off-The-Shelf (COTS) microscope
[pictured on next page]
and an
ACE-T sample module
[pictured later]

Measurement approach – 2/16

Light Microscopy Module (LMM) in the Fluid Integrated Rack (FIR)



LMM in the Closed Position or
Operating Configuration



LMM in the Open Position or
Installation/Service Configuration

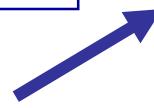
Measurement approach – 3/16

LMM Implementation Philosophy

Philosophy: Maximize the scientific results by utilizing the existing LMM capabilities. Develop small sample modules and image them within the LMM

Light Microscopy Module

Payload specific and multi-user hardware customizes the FIR in a unique laboratory configuration to perform research effectively.



Payload Specific Hardware

- Sample Cell with universal Sample Tray
- Specific Diagnostics
- Specific Imaging
- Fluid Containment

Multi-Use Payload Apparatus

- Test Specific Module
- Infrastructure that uniquely meets the needs of PI experiments
- Unique Diagnostics
- Specialized Imaging
- Fluid Containment

FCF Fluids Integrated Rack

- Power Supply
- Avionics/Control
- Common Illumination
- PI Integration Optics Bench
- Imaging and Frame Capture
- Diagnostics
- Environmental Control
- Data Processing/Storage
- Light Containment
- Active Rack Isolation System (ARIS)

Measurement approach – 4/16



Light Microscopy Module
(LMM)

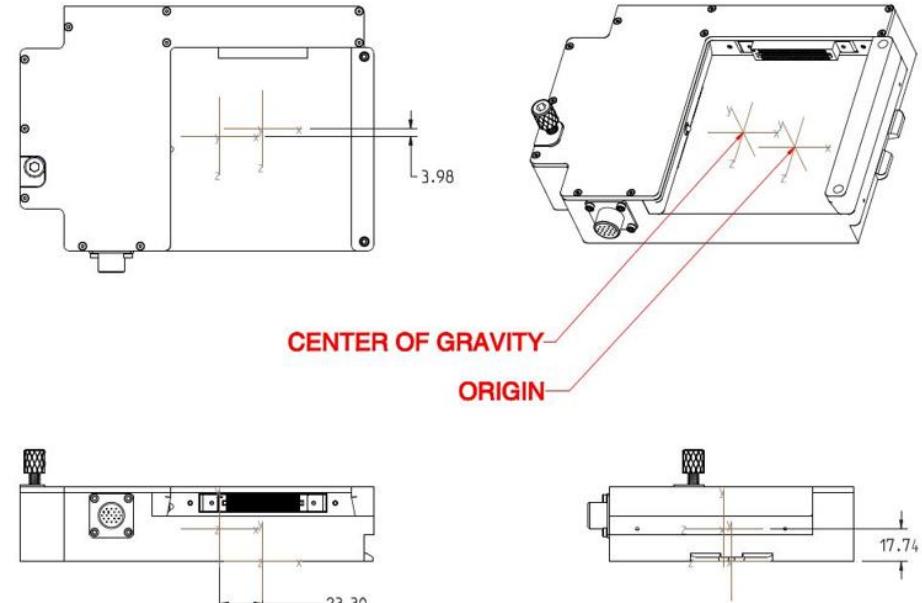
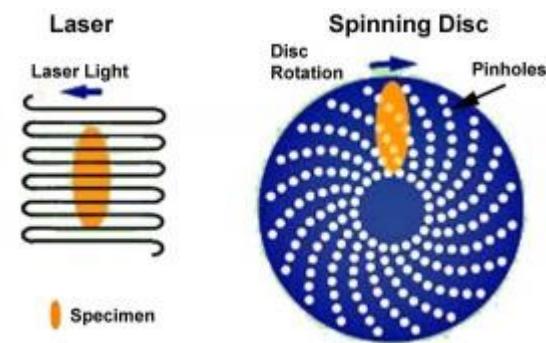
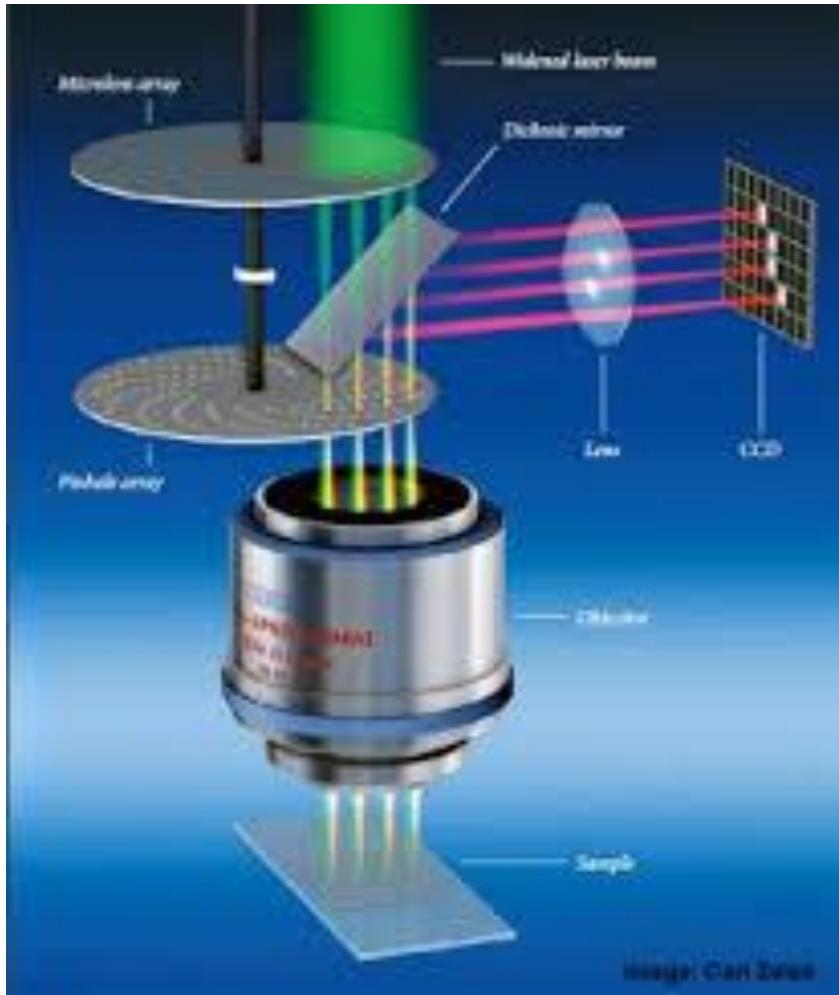


Figure 1: Center of Mass for ACE-T

ACE Sample Assembly with
Removable ACE-T Sample Tray
that will contain a row of 3
temperature controlled
capillary cells

Measurement approach – 5/16



Measurement approach – 6/16

The difference between traditional and confocal microscopy

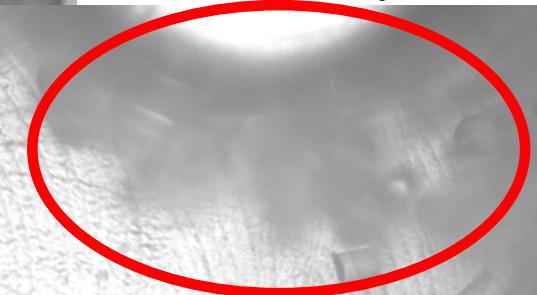
*Traditional microscopy
doesn't see through
objects well; out-of-
focus light obscures
in-focus light*

Focus 50 µm below cover slip



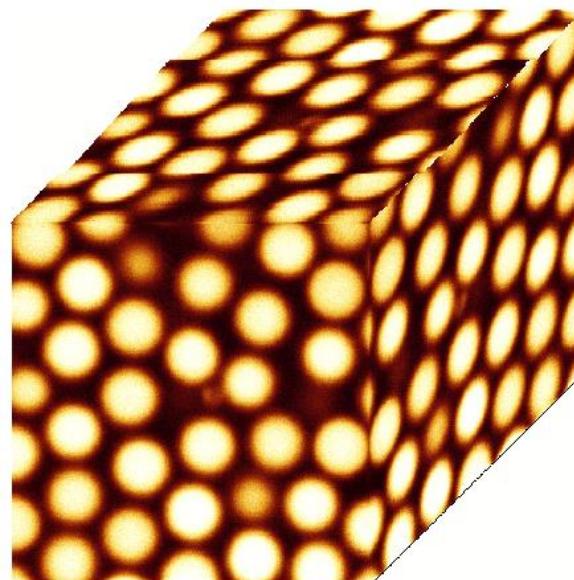
**Particles on top are
easily imaged**

*Focus 200 µm below
(near bottom of well)*



**Particles on bottom
are entirely obscured**

*Confocal microscopy
rejects out-of-focus
light, to look
through semi-
transparent objects*



**3D image is built out of
2D XY sections while
stepping through Z axis.**

Measurement approach – 7/16

ACE-M2R Test Matrix

Experiment Steps:

- 1. Inspect Samples
- 2. Ground to choose first of three sample strips and first Sample to test; feedback to crew.
- 3. Mix all samples in strip using drill BCAT magnet for 30 seconds.
- 4. Apply oil in (Auxiliary Fluids Container) AFC and install assembly. It may require more than one drop of oil be applied when initiating a run of five sample wells on a strip.
- 5. Define XYZ offsets (assembly alignment per ACE-1 method).
- 6. Adjust camera parameters using 2.5x objective and B/S cube.
- 7. Survey well at 2.5x; determine primary (and secondary?) test locations (select locations away from stir bar or bubble).
- 8. Adjust and record best camera parameters using 100x oil objective and Texas Red filter.
- 9. Survey and record best Z-depth at each primary test location.
- 10. Experiment on one well using 100x oil objective; each image set at highest frame-rate (6 to 8 fps), no binning (8 bpp highest supported), full frame images. Store 100 images using Texas Red filter.
- 11. Move to next good sample in strip of 5 samples and Repeat until all good samples in strip have been imaged as outlined above.
- 12. Repeat measurements for all good samples in strip using a time interval of 0, 2, 4, 8, and 16 hours. This means that an entire strip of 5 samples can be run in under 1 ½ days.
- 13. The goal is to successfully image 15 unique sample wells. 15 of the 30 wells are replicates and are flight spares.

Measurement approach – 8/16

- ACE-M2R utilizes one or two sample cell modules, each with 15 sample wells (3 strips of 5 wells each) – the two sample cell modules combine to make a total of up to 30 sample wells. These modules are no longer produced, so unless the on orbit samples are still good [checking on them now], we may not be able to come up with more than 15 sample cells (one sample module).
- Each well holds around 2 microliters of sample fluid
- Two sample cell modules would provide 2 redundant sets of samples.
- All wells contain stainless steel stir bars (magnetic).
- There are two fill ports for each well. The ports are filled with Kalrez® plugs. Summers Optical Cement F-65 is applied over the plugs.

Measurement approach – 9/16

Operational Requirements [nasa.gov]: Defines constraints and requirements necessary to complete the investigation (number of subjects or observations, spacing of observations, downlink of data, return of samples, etc.). **Investigation Summary Form (New Investigations Only – NO PROPRIETARY INFORMATION)**

- One experiment per week; duration of 1-1 ½ days. Each experiment consists of running one strip of 5 samples (due to oil limitations for 100x oil emersion optics). All samples will record 100 frames (at the fastest frame rate, probably 6 fps) at 0, 2, 4, 8, and 16 hours (viewing all samples on a strip sequentially when these time intervals occur). It is understood that the 0 interval may occur and 1 ½ hours or more after mixing when the mixing occurs outside the LMM.
- Run spare Sample Modules if air bubbles are too big. There are 3 strips in each sample cell (of 5 samples each) and 3 backup strips in the spare sample cell (module) to replace runs for samples that have large air bubbles or other problems.
- Between weekly runs, analyze data, re-write scripts, and adjust parameters.
- Number of wells per experiment have historically (*i.e.*, ACE-M2) been limited by:
 1. Data bottlenecks on IPSU and IOP
 2. XY position repeatability (need to return to the same particle set or don't move during experiment => one well position. This takes too long, so find a solution. Images can be registered in post-processing via port or stir bar location, or pattern of particles stuck to bottom of cover slip.
 3. Oil availability – available immersion oil limits runs to one strip at a time; air objective has no such constraints.

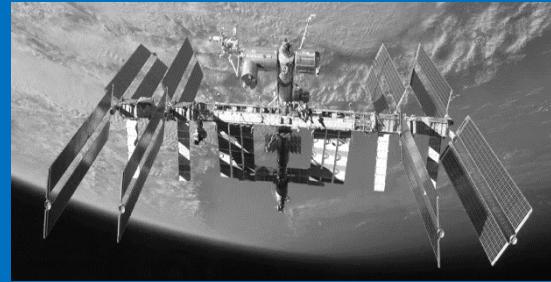
Measurement approach – 10/16

Operational Protocols [nasa.gov]: Descriptive overview of the investigation on orbit procedures.

Experiment Steps:

1. Inspect Samples
2. Ground to choose first of three sample strips and first Sample to test; feedback to crew.
3. Mix all samples in strip until you get the consistency previously obtained using the drill BCAT magnet for 30 seconds.
4. Apply oil in (Auxiliary Fluids Container) AFC and install assembly. It may require more than one drop of oil be applied when initiating a run of five sample wells on a strip.
5. Define XYZ offsets (assembly alignment per ACE-M2 method).
6. Adjust camera parameters using 2.5x objective and B/S cube.
7. Survey well at 2.5x; determine primary (and secondary?) test locations (select locations away from stir bar or bubble).
8. Adjust and record best camera parameters using 100x oil objective and Texas Red filter.
9. Survey and record best Z-depth at each primary test location.
10. Experiment on one well using 100x oil objective; each image set at highest frame-rate (6 to 8 fps), no binning (8 bpp highest supported), full frame images. Store 100 images using Texas Red filter.
11. Move to next good sample in strip of 5 samples and Repeat until all good samples in strip have been imaged as outlined above.
12. Repeat measurements for all good samples in strip using a time interval of 0, 2, 4, 8, and 16 hours. This means that an entire strip of 5 samples can be run in under 1 ½ days.
13. The initial goal was to successfully image 15 unique sample wells [now 9 unique sample wells with the limited number of sample cells available to fill, unless the ACE-M2 samples on orbit are still good]. 15 [now 4] of the 30 [now 15] wells are replicates and are flight spares.

Importance and reason for ISS



On Earth, gravity plays a big role in how liquids and gases interact: The heavier liquid phase settles beneath a lighter gas phase, and these differences in density enable easy separation. But the flow and behavior of complex fluids and phase separation where there is little or no gravity is not well understood.

A greater understanding of liquid-gas interaction in microgravity could benefit a wide range of fluid storage, transport and processing systems for future spacecraft.

The Earth benefits are will compliment the ACE-T6 (Lynch – P&G) work on stabilizers. The ACE-T6 work focuses on the effects of polydispersity on stabilizers. ACE-M2R focuses on monodisperse colloidal particles, which are more fundamental and easier (possible) to model with theory. ACE-T6 dwells on both science and engineering (seeing what works when real-world “dirty” systems that are too complicated to model. See the next slide for more “down to Earth” reasons about the importance of the ACE-M2R work.

Expected results and how they will advance the field

- This work with colloids is looking at a “clean” monodisperse model system (containing a single size colloid suspended in known ranges of depletant concentrations) to find and adjust the theoretical foundations.
- As shown in an earlier slide (above), some unexpected results were seen (phase separation was still observed) in BCAT and ACE-M2 experiments after the critical point was “passed”. So something needs to be adjusted, the model or how it is implemented.
- Reflying ACE-M2 with confocal should provide MUCH clearer images that are now 3D - from which to extrapolate.
- ACE-M2R is an case where looking at details should enable us to see the big picture.

Earth benefits/spin-off applications

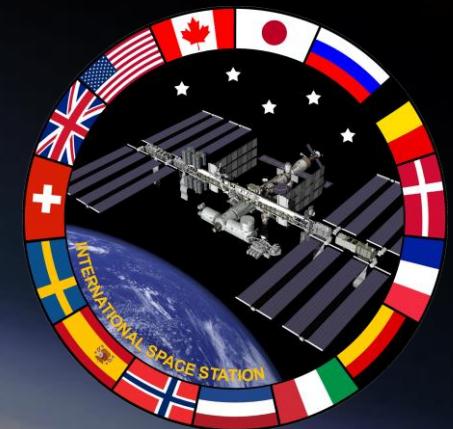
- The knowledge of phase separation and gelation is important to the development of consumer and household products produced by industry, including large corporations like Procter & Gamble, with whom this science team is collaborating on a number of joint efforts, with billions of dollars in sales each year of signature products. These products are largely based on complex fluids, with components very similar to, and obeying the same physics, as the model system materials we are flying with ACE. Quantitative and qualitative insights from previous ISS and ground-based experiments have assisted them in designing formulations to maximize stability and shelf-life. By providing information and insight into the fundamental physical processes at work, without interference from gravity aboard ISS, this work has given them the understanding and tools that enable them to design better consumer products when gravity is important, on Earth. The detailed microscopic understanding that we anticipate to come from our ACE experiments will provide them with even more specific data and quantifications, providing an improved understanding for the development of their products, including those worth billions of dollars annually to the US economy.



ACE-M2R

Increment 53/54 Science Symposium

BACKUP SLIDES





Samples 1 – 5 for ACE-M2R

Description: HARVARD ACE Engineering Sample #1

P/N: PLU1

Composition: 27.1% colloid particles in solvent mixture.

Description: HARVARD ACE Engineering Sample #2

P/N: PLU2

Composition: 13.6% colloid particles in solvent mixture.

Description: HARVARD ACE Engineering Sample #3

P/N: PLU3

Composition: 6.24% colloid particles in solvent mixture.

Description: HARVARD ACE Engineering Sample #4

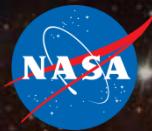
P/N: PLU4

Composition: 2.79% colloid particles in solvent mixture.

Description: HARVARD ACE Engineering Sample #5

P/N: PLU5

Composition: 2.29% colloid particles in solvent mixture.



Samples 6 – 9 for ACE-M2R

Description: HARVARD ACE Engineering Sample #6

P/N: PLU6

Composition: 1.13% colloid particles in solvent mixture.

Description: HARVARD ACE Engineering Sample #7

P/N: PLU7

Composition: 0.52% colloid particles in solvent mixture.

Description: HARVARD ACE Engineering Sample #8

P/N: PLU8

Composition: 0.33% colloid particles in solvent mixture.

Description: HARVARD ACE Engineering Sample #9

P/N: PLU9

Composition: 0.22% colloid particles in solvent mixture.

[Some of the above samples well will be duplicated into remaining spare wells.]

All particles are spheres of polymethylmethacrylate (PMMA) with a Cy3-MMA dye, suspended in a solvent mixture that is 18% cis-decalin, 22% tetralin and 60% tetrachloroethylene (by mass). All of the materials for these samples have been approved, launched and imaged as part of the ACE-M2 and BCAT-KP experiments onboard the International Space Station.



ACE-M2R Success Criteria

Success Level	Accomplishment
Minimum Success	<ol style="list-style-type: none">1. Observe at least one of each functional launched colloidal suspension sample at low (air) and high-magnification (oil immersion), at multiple locations and high speed, in order to allow quantification of spatiotemporal resolution of the microscope, with the goal of using the wide-field fluorescence DDM analysis technique to measure the particle diffusion constant.2. Observe at least one of each functional launched colloid-polymer sample at low and high magnification, at several points in space, and at the same point over time, to assess time evolution in phase-separation and related processes in these samples.
Significant Success	Perform the above measurements for all functional launched samples, which include several wells of the same original sample, to assess reproducibility in the loading, launch and imaging processes, and any effects on resulting sample behavior.
Complete Success	Repeat all measurements as defined under "significant success" several times, to assess reproducibility of the behavior in the same samples over time.



Microgravity Justification

- Formation of colloidal clusters is profoundly affected by gravity via sedimentation processes. Chaikin and Russel have already demonstrated this effect in space experiments exploring the simplest of all entropic transitions, the hard-sphere liquid-solid phase transition.
- Sedimentation causes particles to fall so rapidly that there is insufficient time for particles to explore the full phase space of positions and velocities that are required for thermodynamic assembly processes. A substantial particle concentration gradient arises in the earthbound sample.

$$h = \frac{k T}{\Delta \rho V g}$$

h = gravitational height

$k T$ = Thermal Energy of system

$\Delta \rho$ is the density difference between the particles and the background fluid

V is the particle volume

g is the gravitational acceleration

h ranges from a few microns for the case of polystyrene in water to a fraction of a micron for most of the other particles we consider. Our particles are usually of order 1 micron in diameter.



ACE-M2 - getting ready to celebrate New Years

